A New Approach to Customized Atomic Absorption Analysis

Application Note

Atomic Absorption

Authors

D. L. Hoobin and P.R. Liddell,

Introduction

For several years, it has been possible to control atomic absorption spectrophotometers (and many other analytical instruments) from small computers. By far the most common programming language has been Basic but, in most cases, the Basic language and the computer have been designed for general purpose applications. Interfacing to instruments has been complicated and the computer has often been limited to gathering and manipulating results. In the few cases where instrument control has been possible, it has been done by simulating key presses on the instrument keyboard. Applications which are not normally catered for in routine measurements, but might be required in a customized system, include unusual report formats, plotting of long term trends, ratioing concentrations and automatic optimization of flame or furnace parameters.

The Agilent SpectrAA-30 and SpectrAA-40 atomic absorption spectrophotometers provide for such applications by allowing complete control of all instrument parameters using a much-enhanced Basic language. Interfacing is straight-forward and the instrument and accessories (sample changer or graphite furnace) can be controlled from the same program.

The DS-15 data station forms an integral part of the SpectrAA-30/40 system. The whole system is controlled from its keyboard and video display. The DS-15 performs most of the signal processing and instrument control tasks normally done within the spectrophotometer and it is also able to carry out the extended data processing tasks which normally require an additional computer. Although the DS-15 may be used as a general purpose computer or be interfaced to other instruments, the Extended Basic functions described in this paper are only available with the SpectrAA-30 or SpectrAA-40.

Programs in Basic are not required for routine instrument operation. The normal user interface is through a series of video screen pages which utilize a simple “fill-in-the-form” (FITF) approach to parameter entry. This interface is written in the

programming language, Forth. Figure 1 shows the Instrument Parameters page. Parameters are selected by positioning the cursor and values are entered from the keyboard. The FITF interface provides all the instrument set-up, optimization, calibration, graphics, automatic multi-element analysis and report generation features normally required. However if the user has a specific or unusual requirement not satisfied by FITF then this can be implemented through the use of the system's Extended Basic. The Extended Basic provides completely open ended flexibility for instrument control and data processing.

```
PROGRAM 1            Cr  5 ppm in 1% HCl
BLANK                REPLICATE    1
ABSORBANCE           EXPANDED BY 1    BC ON
INTEGRATION          1.0      (sec)   AIR-ACETYLENE
PRINTING
                 INSTRUMENT PARAMETERS

LAMP POSITION                1
LAMP CURRENT (mA)            7
SLIT WIDTH (nm)              0.2
SLIT HEIGHT                  NORMAL
WAVELENGTH (nm)              357.9
FLAME                        AIR-ACETYLENE
SAMPLE INTRODUCTION          AUTO NORMAL
DELAY TIME (sec)             0
MEASUREMENT TIME (sec)       1.0
REPLICATES                   1
BACKGROUND CORRECTION        ON
EXPANSION FACTOR             1
```

Figure 1. Instrument parameters page.

All instrument and accessory parameters are accessible from the Extended Basic through a software interface termed the Instrument Operating System (IOS). Each parameter is assigned a location in a data dictionary. Examples of such parameters are wavelength, sampler carousel position, initiation of a reading, gas flows and graphite furnace temperatures. Any parameter can be set from Basic by using a single Deposit statement containing the appropriate data dictionary location and parameter value. Similarly the value of any parameter can be read into the Basic program by using a single Recall statement. Hence complex instrument control tasks are reduced to a sequence of Deposit and Recall statements to and from the data dictionary. The entire contents of the data dictionary may be stored or recalled as a self contained analytical program.

A brief description of the software components involved will be given and examples of the Extended Basic commands will be explained. Finally the results of a program written using the Extended Basic toinvestigate automated flame optimization will be presented.

System Software Components

Figure 2 shows the main software components and their interactions when controlling the SpectrAA-30/40 from the Extended Basic. The arrows indicate the flow of control information, data flow is in the reverse direction. When FITF is used, it replaces Basic in Figure 2.

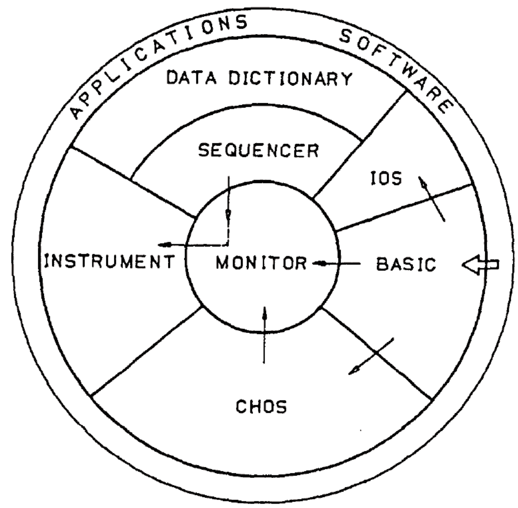

Figure 2. Enhanced Basic software srchitecture.

Figure 3 illustrates the flow of control that occurs when an Extended Basic command is executed. All the user sees is the command being issued and results or errors being returned, the details of the processes involved are hidden by the IOS interface.

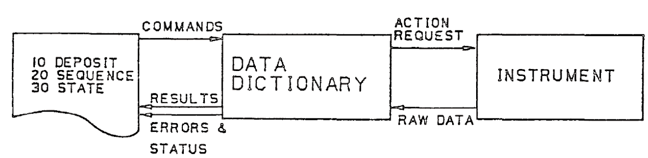

Figure 3. Instrument control from enhanced Basic.

Monitor: This comprises a collection of low level functions for controlling the DS-15 keyboard, screen and interconnecting data bus. It resides permanently in the DS-15 PROM (Programmable Read Only Memory), whereas all other components reside on disk and are loaded into RAM (Random Access Memory) on power-up.

Chos: The CHannel Operating System is the disk operating system and channel manager. A channel may be a disc file or device (printer, modem, analytical instrument).

Sequencer: This is an interpreter which, running as a background task, initiates and controls instrument and accessory actions in real time.

Data Dictionary: The data dictionary is an ordered collection of data and its associated properties. The applications programs access the data via numeric identifiers, termed Nodes. The contents of the data dictionary reflect the current operational state of the instrument and accessories. The data may be recalled or modified by the user via the Extended Basic commands. Modifying data will cause a corresponding modification to the state of the instrument or accessory.

IOS: The Instrument/Operating System is an interface between the data dictionary and FITF or Basic. It contains the functions which manipulate the data dictionary.

Table 1. Some Examples of Nodes in the Data Dictionary

Node	Name	Values	Interpretation
06	Instrument mode	0	Absorbance
		1	Flame emission
		2	Lamp emission
39	Lamp current	0-20	milliamps
48	Probe position	0	Up
		1	Down
79	Acetylene flow	1.5-8.0	litres/min

Applications Program

- Normal Operation: The normal user interface is through the Fill In The Form (FITF) pages. An optional Utilities package provides a number of Extended Basic programs written by Varian. The Utilities perform data editing, report generation, weight correction and data archiving. They can be easily listed and used as examples or guides for users developing their own customized applications.
- Extended Operation: The Utilities package provides access to the Extended Basic. New programs can be incorporated into the existing Utilities Menu shown in Figure 4, with the Install utility.

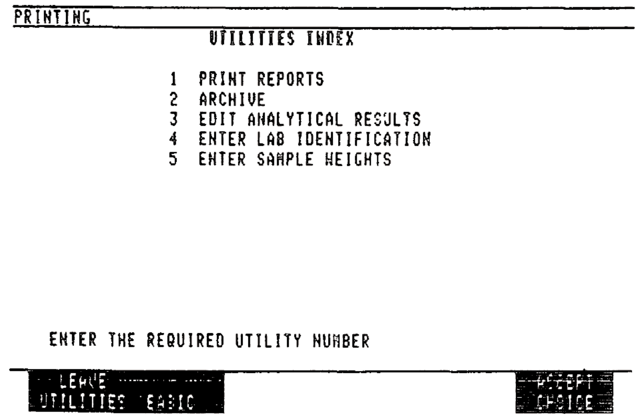

Figure 4. Utilities index.

Details of Enhanced Basic

The Enhanced Basic contains new reserved words, such as Deposit, Recall, Sequence which allow the programmer to control the SpectrAA instrument or accessory such as a graphite tube atomizer or sample changer. The general idea is that the user program may store or recall data from the data dictionary and the storing or recalling of that data may perform some instrument hardware related action.

Once a particular instrument/accessory configuration has been established in the data dictionary, the entire contents may be stored on, or recalled from disk using the Program Store and Program Load commands.

The following simple programs illustrate how the Deposit, Recall and Sequence statements, together with a knowledge of the data dictionary nodes, may be used to execute very diverse tasks.

Example 1

This program drives the programmable sample changer to the blank tube position, drops the probe into the solution and performs a zero on the resulting signal.

```
10 Deposit 48;1          Drive probe down when at position
20 Deposit 47;1          Set sample type to blank
30 Sequence 13           Drive sampler to blank
40 If busy(0) then 40    Wait for sequencer to complete action
50 Sequence 14           Perform a zero on data signal
60 If busy(0) then 60    Wait for sequencer to complete action
```

Example 2

This program selects absorbance mode, sets the read time to 1 Sec and takes a reading.

```
10  Deposit 6;0          Select absorbance mode
20  Deposit 10,0;1       Set 1 second measurement time
30  Deposit 1;0          Take a reading
40  If busy(1) then 40   Wait for read time to expire
50  Recall 4,0;result    Get result from data dictionary
60  Print result         Display reading
```

Example 3

This program configures the flame control unit to air-acetylene and sets the gas flows for fuel and oxidant.

```
10  Deposit 75;1         Select air-acetylene flame type
20  Deposit 77;13.5      Set air flow to 13.5 liter/min
30  Deposit 79;3         Set acetylene flow to 3 liter/min
40  Sequence 17          Setup entire system
50  If busy(0) then 50   Wait for sequencer action to finish
```

Flame Optimization Example

This program was written in Extended Basic to investigate automated optimization of fuel flows. Figure 5 shows the parameter entry page where the operator enters values for maximum and minimum acetylene flow, fuel flow increment and oxidant flow. This page is constructed in Basic to match the normal FITF protocol.

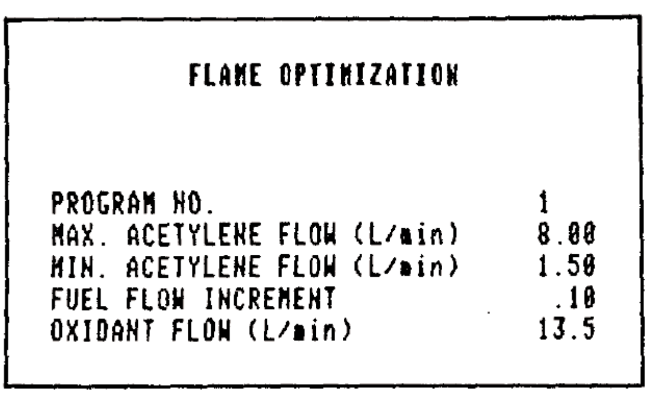

Figure 5. Flame optimization parameter entry.

The software sets the programmable flame control unit to the minimum acetylene flow then drives the sample changer to the blank and reslope positions, taking a reading at each position. The difference in absorbance is then plotted against acetylene flow.

The acetylene flow is incremented by the amount specified and the process is repeated until the maximum flow is reached. On reaching the maximum flow the sampler is driven to the rinse position and the fuel flow corresponding to maximum absorbance is entered into the data dictionary.

A graph of absorbance versus fuel flow is automatically plotted on the screen and may be printed out.

Results

Chromium in the Air/Acetylene Flame.
5 mg/L Chromium in 0.2M HCI was aspirated and the acetylene flow was changed from 2 to 5 L/min with a flow increment of 0.1 L/min. The air flow was fixed at 13.5 L/min. This was repeated for a number of burner heights. The maximum absorbance was obtained with a burner height of 5mm and an acetylene flow of 3.7 L/min. The plot for burner height of 5mm is shown in Figure 6. Figure 7 shows that the optimum acetylene flow and maximum absorbance both decrease when the burner is raised to a height of 10mm.

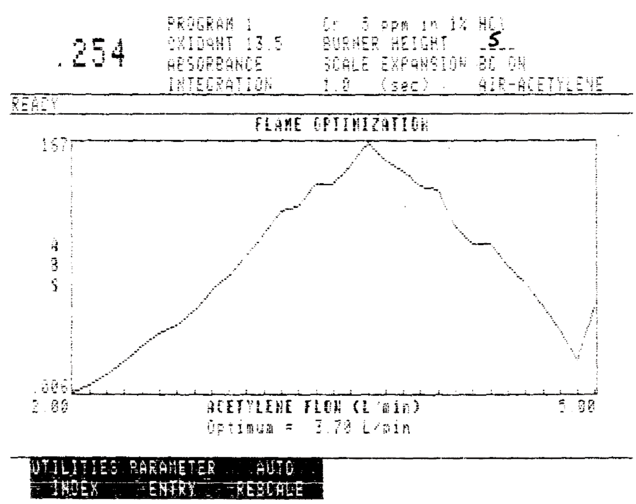

Figure 6. Cr, burner height = 5 mm.

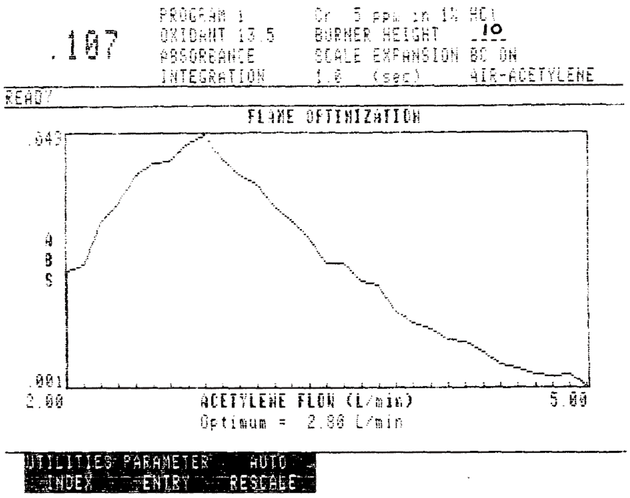

Figure 7. Cr, burner height = 10 mm.

Calcium in the N20/Acetylene Flame.
0.5 mg/L Calcium in 0.1 M HCI was aspirated and the acetylene flow was changed in the first instance from 4 to 10 L/min with a flow increment of 0.1 L/min. The nitrous oxide was kept fixed at 11.0 L/min. This was repeated, changing the acetylene flow from 6 to 8.5 L/min, enabling a more detailed examination of the signal around the optimum fuel flow. The burner height remained fixed at 1 mm for these measurements. The plots shown in Figures 8 and 9 show that the optimum fuel flow for calcium is 7.3 L/min.

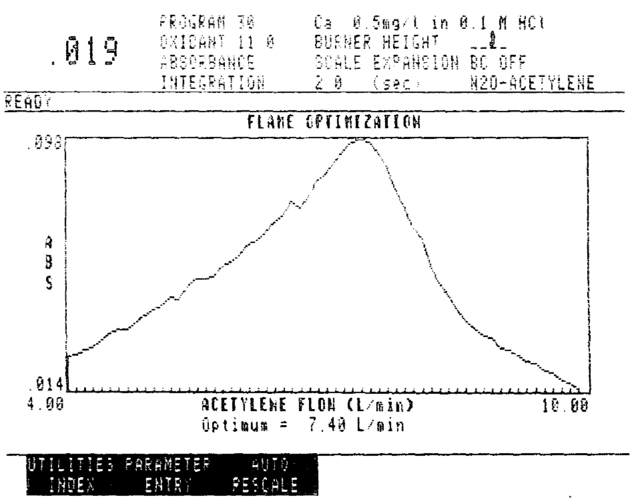

Figure 8. Ca, acetylene range 4–10 L/minute.

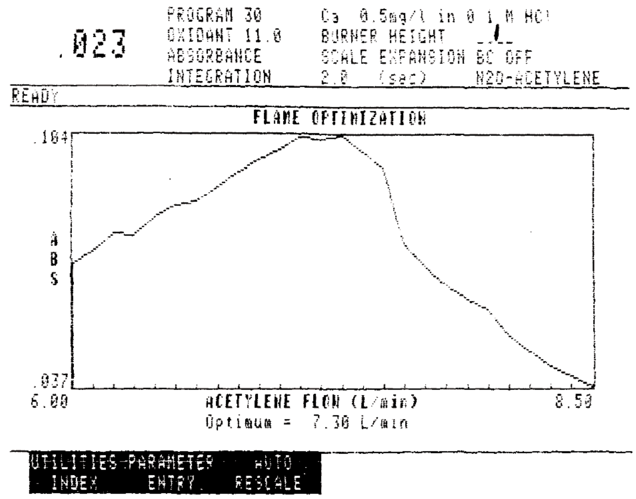

Figure 9. Ca, acetylene range 6–8.5 L/minute.

Conclusion

The SpectrAA-30/40 enhanced BASIC language provides a powerful set of tools for controlling instrumental hardware and manipulating data to solve problems not normally catered for in routine measurements.

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